FABRICATION OF HOLOGRAPHIC OPTICAL ELEMENTS FOR THE NEAR INFRA-RED BASED ON POLYMER MATERIALS

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ABSTRACT

Holographic Optical elements (HOE's) such as Holographic filters and Holo-lenses have been fabricated in Methylene blue sensitized Gelatin (MBG) and Dichromated polyvinyl alcohol (DCPVA). Holographic filters fabricated in MBG exhibit high diffraction efficiency (≈90%) at low exposure energy of ≈46 mJ/cm^2, while Holo-lenses fabricated in DCPVA have ≈60% diffraction efficiency.

2.INTRODUCTION

The use of HOE's in grating monochromators, laser scanners, head-up displays, optical interconnects, fiber couplers, matched filters is well established. The performance of these devices depends to a large extent on the efficiency of the recording media. It is well known that dichromated gelatin (DCG) is one of the best recording medium for the fabrication of HOE's (1-3). However it suffers from limited spectral sensitivity (upto 500 nm), low environmental stability and rigorous wet processing procedures. To overcome these problems related to wavelength sensitivity of DCG and wet processing we have investigated two materials viz., Methylene blue sensitized gelatin (MBG) and Dichromated polyvinyl alcohol (DCPVA).

2.1 *METHYLENE BLUE SENSITIZED GELATIN (MBG)*

Various researchers have worked on the sensitization of DCG for the red region by incorporating dyes such as methylene blue (4-8). But the energy sensitivity remained low (≈500 mJ/cm^2) for diffraction efficiency of 80%. Also, the solubility of methylene blue

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dye in higher concentrations is a problem in the presence of dichromate in Methylene blue sensitized dichromated gelatin (MBDCG). This problem has been overcome by Solano et.al. (9) by using the Methylene blue sensitized gelatin (MBG) process, where the methylene blue dye is first photoreduced and later fixed by a dichromate solution. But the energy required to obtain ≈ 70% diffraction efficiency was in the range of 500-600 mJ/cm^2. The energy sensitivity could be slightly improved by carrying out the photoreduction in an inert atmosphere as shown by Capolla et.al. (10).

Efforts have been made to improve the energy sensitivity of MBDCG comparable to that of DCG by introducing electron donors to assist the photoreduction of the dye (4,7,11), but this did not significantly reduce the exposure energy required to achieve maximum diffraction efficiency. Jeff Blyth (12) employed tetramethylguanidine (TMG) as an electron donor in conjunction with methylene blue and could produce bright reflection holograms (value of diffraction eficiency not quoted) recorded at 633 nm at an exposure level of 50 mJ/cm^2. Here we present the results of our experimental study on a new Methylene blue sensitized gelatin/ electron donor system with higher energy sensitivity (≈46 mJ/cm^2) and its use for the fabrication of holographic filters both in the transmission and reflection mode.

2.2 *DICHROMATED POLYVINYL ALCOHOL (DCPVA)*

In the past decade, research in the field of holographic recording materials have been geared towards real-time developing, dry process media. In our research endeavours we have investigated a few polymer based media such as Dichromated polyvinyl alcohol (DCPVA) (13), Feric chloride PVA (FePVA) (14) and Dichromated polyacrylic acid (DCPAA) (15). Here we present the results of our study with DCPVA and its use in the fabrication of Holo-lenses.

Dichromated polyvinyl alcohol (DCPVA) is sensitive in the blue green region of the spectrum and when used for recording holograms the holograms develop in real time. This characteristic of the media enables us to record holograms with 488 nm or 514 nm wavelength of the Argon laser and monitor the development using the 633 or 647 nm wavelength of the He-Ne or Krypton laser.

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Photochemical reactions of DC-PVA have been followed by infrared spectroscopy (16) and the conclusions drawn from these are: (1) PVA undergoes oxidation and the hydroxyl group disappeared with the formation or increase of carbonyl (-C=O) and carboxylic (-COOH) groups. (2) Cr(VI) reduced to Cr(III) which has been identified from the disappearance of characteristic absorption of the NH- and $Cr_2O_7^{2-}$ groups. (3) It has been suggested that the carbonyl (-C=O) and carboxyl groups (-COOH) of PVA undergo co-ordination with the trivalent chromium.

Based on these findings a detailed experimental study on the behaviour of DC-PVA under polarized light illumination for real-time polarization holography has been carried out (17). However, the final efficiency of the holograms after post exposure heat treatment was only 30% (17,18). Here we present our results on the improvement of the diffraction efficiency of transmission holograms.

3.RESULTS AND DISCUSSION

3.1. *Holographic Filters in Methylene Blue sensitized gelatin*

Volume reflection holograms in DCG because of their high efficiency, low scatter and narrow spectral bandwidth can be used as wavelength filters. Such filters play a major role in expanding the bandwidth capacity of future fiber communication and local area network systems. Duncan et.al. (3) have fabricated narrow band holographic reflection filters in DCG that are linearly tunable over 1.2 to 1.6 μm wavelength region and have been used as a single channel selector for wavelength-multiplexed optical fiber systems. While, Moslehi et.al. (19) have reported theoretical and experimental results of novel fiber optic wavelength-division multiplexing (WDM) design employing a broad band (> 150 nm) DCG volume holographic grating operating in a reflective littrow configuration with on-axis optics, a single lens and one fiber array.

To shift the wavelength sensitivity of DCG various methods have been used. A comparison of the various studies done so far is presented in Table 1. Among these the simplest method is that of Refs.9 & 10, which also avoids the problem of precipitation of methylene blue in the presence of dichromate salt. The photochemical study of the real-time bleaching of MBG (20), gave a better understanding of the recording mechanisms

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which led us to the conclusion that a proper electron donor was necessary to increase the energy sensitivity of MBG. Among the various electron donors investigated to achieve this goal two of them viz., Dimethylamino-ethyl-methyl-amino-ethanol (DEMAE) and Dimethylethanol-amine (DMEA) gave excellent results. A ten fold increase in the energy sensitivity resulted (21).

Table 1. Maximum diffraction efficiency (η_p) obtained with different methods of sensitization of DCG for the red region.

AUTHORS	MAXIMUM DIFFRACTION EFFICIENCY (%)	EXPOSURE ENERGY (mJ/cm^2)
Graube (1978)	80	500
Sasaki et.al. (1978)	80	100
Kubota and Ose (1979)	80	100
Solano et.al. (1987)	73	500
Capolla & Lessard (1988)	75	520
Changkakoti & Pappu (1989)	80	500
Capolla & Lessard (1990)	80	48
Jeff Blyth (1991)	?	50

3.1.1 *Transmission gratings in Methylene blue sensitized gelatin/dimethylethanol-amine (MBG/DMEA) system*

Preparation and processing procedures of MBG/DMEA films are given elsewhere (10,22). MBG films 20 to 25 μm thick were used for the holographic characterization. The experimental setup to record transmission and reflection holograms was so arranged so as to record volume holograms. Since the MBG plate is sensitive in both 633 nm and 647 nm, holograms have been recorded in both these wavelengths using He-Ne and Krypton Lasers. The stability of the set up has been controlled using a fringe locking system.

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The diffraction efficiency (η) versus exposure energy plots of transmission gratings recorded with 633 nm and 647 nm wavelengths are presented in Figs.1 and 2

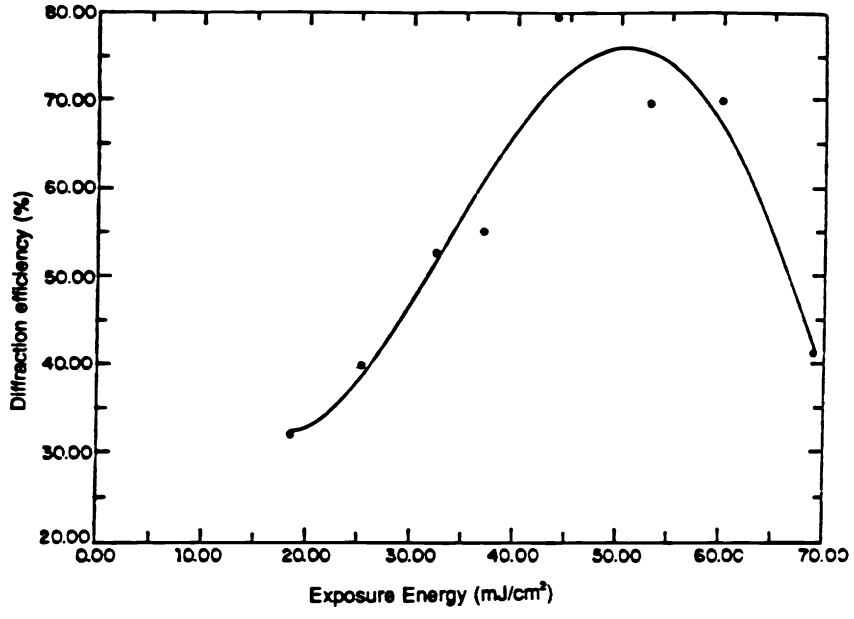

Figure 1. Diffraction efficiency as a function of exposure energy for a MBG/DMEA transmission grating recorded with 633 nm line of the He-Ne Laser.

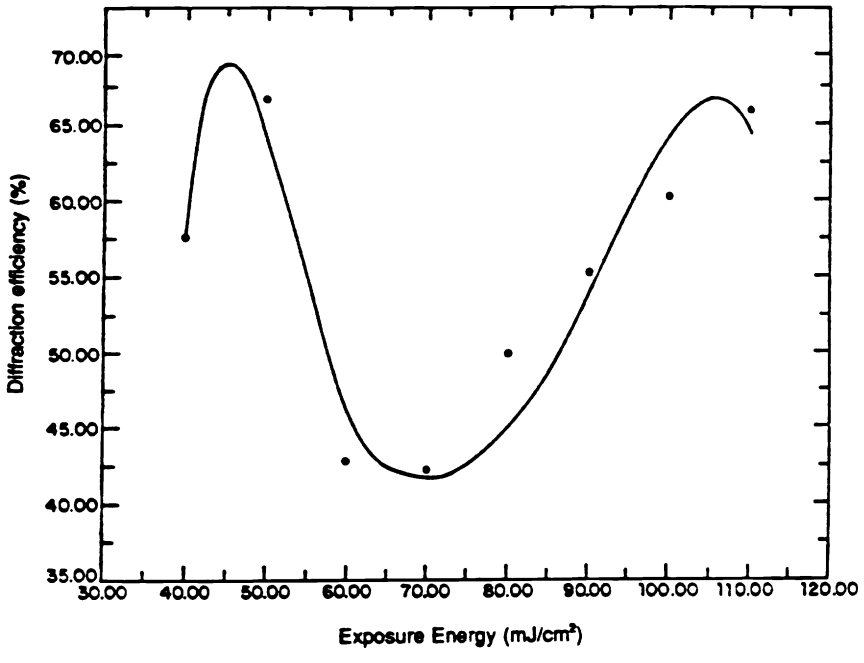

Figure 2. Diffraction efficiency as function of exposure energy of MBG/DMEA transmission grating recorded with 647 nm line of the Krypton Laser.

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respectively. Maintaining the interbeam angle $2\Theta=50°$ the frequency of the gratings recorded with 633 nm is 1355 cycles/mm and 1306 cycles/mm for gratings recorded with 647 nm. In the Figures the solid line represents a polynomial fit of the experimental data (given by dots). The oscillatory behaviour of η (see Fig.2) is in good agreement with Kogelnik's theory (23). The maximumum diffraction efficiency (η_p) value is obtained at exposure energy of 49 mJ/cm^2 for 633 nm and 46 mJ/cm^2 for 647 nm respectively. Thus comparing the two plots it is seen that the energy sensitivity of MBG/DMEA system is almost the same at 633 and 647 nm wavelengths. Also, comparing the values of exposure energy obtained by earlier workers (see Table 1) it is seen that there is almost a ten fold increase in the energy sensitivity with the MBG/DMEA system.

3.1.2 *Reflection gratings in MBG/DMEA system*

Reflection gratings have been recorded in MBG/DMEA films using 647 nm wavelength from the Krypton laser. The interbeam angle was so adjusted so as to obtain a frequency of the gratings as 2532 cycles/mm. The η versus exposure energy curve is presented in Fig 3. It can be observed that diffraction efficiency of 90% can be achieved

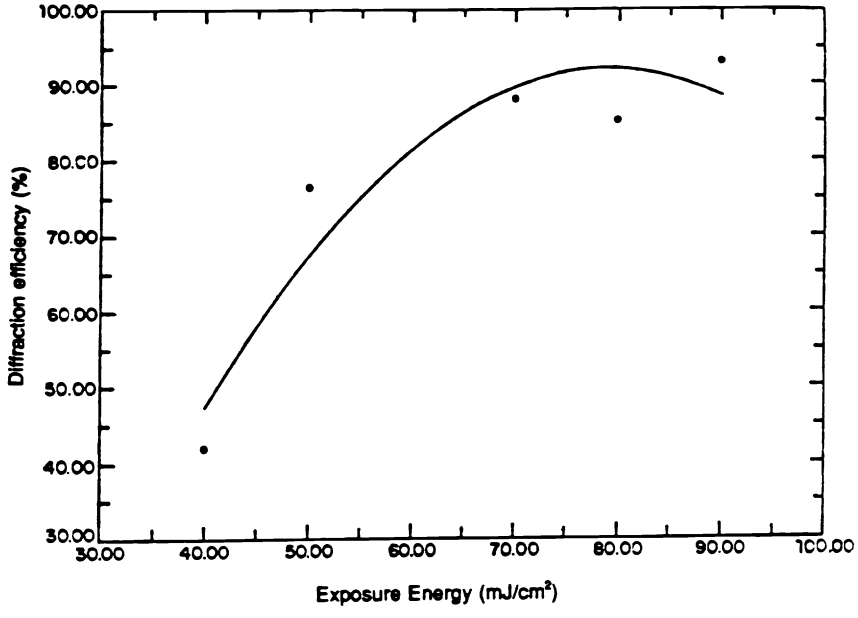

Figure 3. Diffraction efficiency as a function of exposure energy for a reflection grating recorded in MBG/DMEA film with 647 nm line of the Krypton Laser.

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at an exposure energy of 80 mJ/cm^2 while 75 % at an exposure of 50 mJ/cm^2.

3.1.3 *Angular selectivity of the MBG/DMEA filters*

In figure 4. the angular selectivity curve for a transmission grating with a frequency of 1638 cycles/mm recorded in MBG/DMEA film is presented. For convenience (1-η) values are plotted as a function of the angle of incidence. The angular bandwidth of the grating is 3.2° which indicates that the effective thickness of the grating is 17 μm in a film thickness of 24 μm. The narrow angular selectivity of the gratings would enable us to multiplex large number of gratings in the volume of the film.

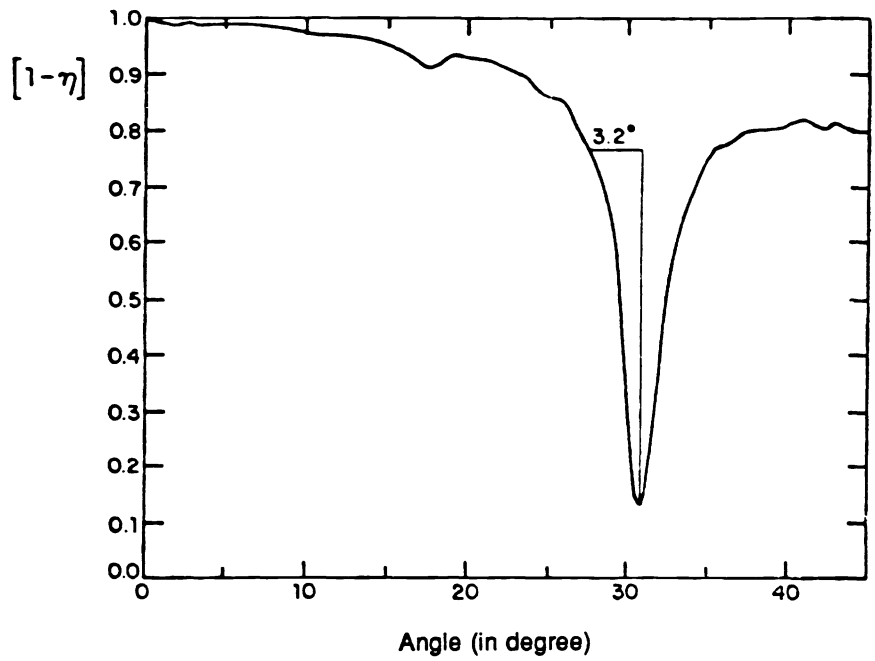

Figure 4. Angular selectivity curve of transmission grating in MBG/DMEA film having a frequency of 1638 cycles/mm.

3.1.4 *Wavelength selectivity of the filters*

To determine the wavelength sensitivity of the transmission filters holographic gratings were recorded with exposure energy of 80 mJ/cm^2. A shift in the wavelength of reconstruction was achieved by varying the temperature of the developing bath. Three temperature were chosen viz., 35°, 37°, and 40°C. The transmittance of these filters as

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function of wavelength are plotted in Figs.5 & 6. This was carried out using a spectrophotometer. It is observed that the wavelength of reconstruction shifts from 600 nm to 656 nm when temperature of the developing bath is varied from 35° to 40°C. This

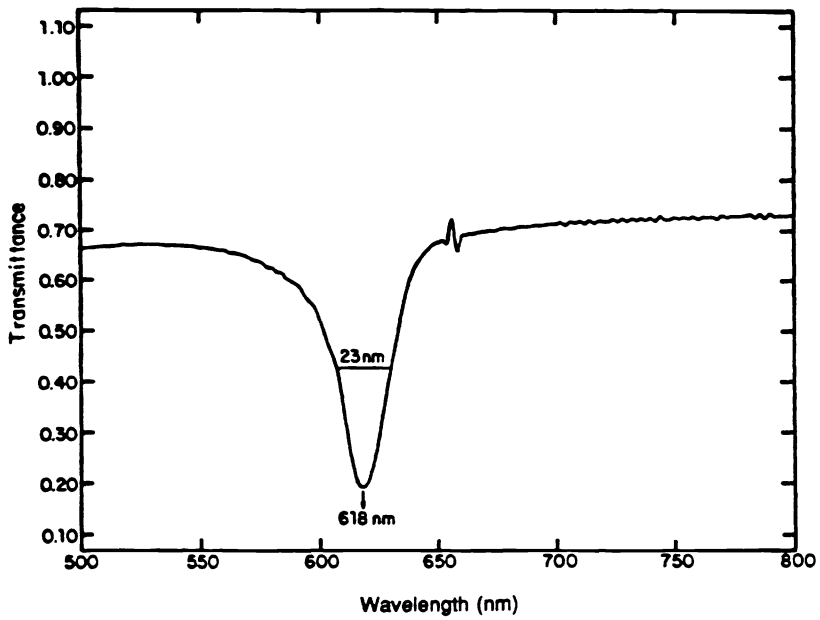

Figure 5. Transmittance as a function of wavelength of transmission filter developed at 37°C.

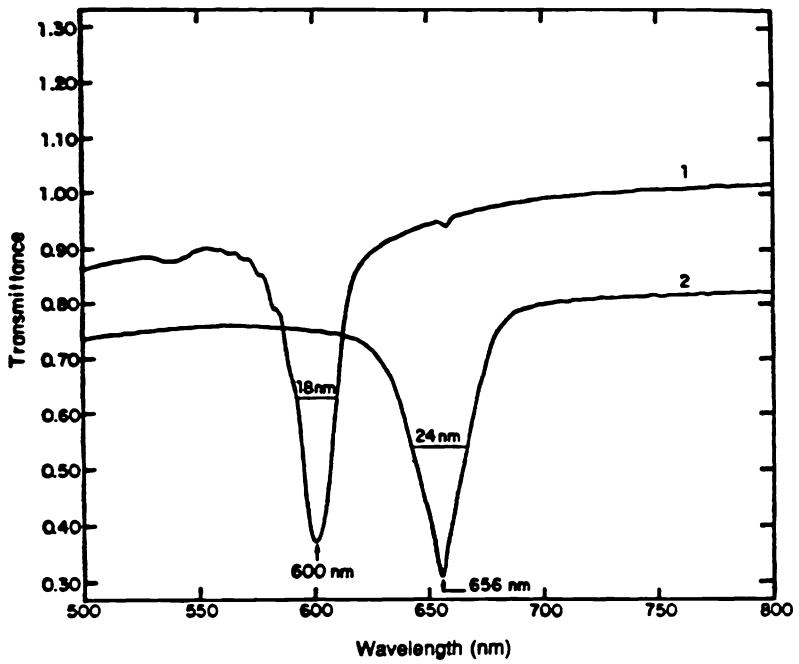

Figure 6. Transmittance as a function of Wavelength for two transmission filters developed at 35°C and 40°C respectively.

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interesting result gives us the possibility of fine tuning of such filters to the required wavelength of reconstruction. However, the bandwidth increases from 18 nm in the case of the holograms developed at 35°C to 24 nm in the case of one developed at 40°C.

In the case of filters in the reflection mode the shift in the reconstruction wavelength is achieved by varying the geometry of the recording setup. The transmittance versus wavelength of such filters are presented in Figs 7. In Fig 7 the curves represent those for reflection filters recorded at 130 mJ/cm^2. Their wavelength of reconstruction is at 800 nm. These divices can be very useful for fabrication of wavelength division multiplexers and demultiplexers for fiber to fiber coupling.

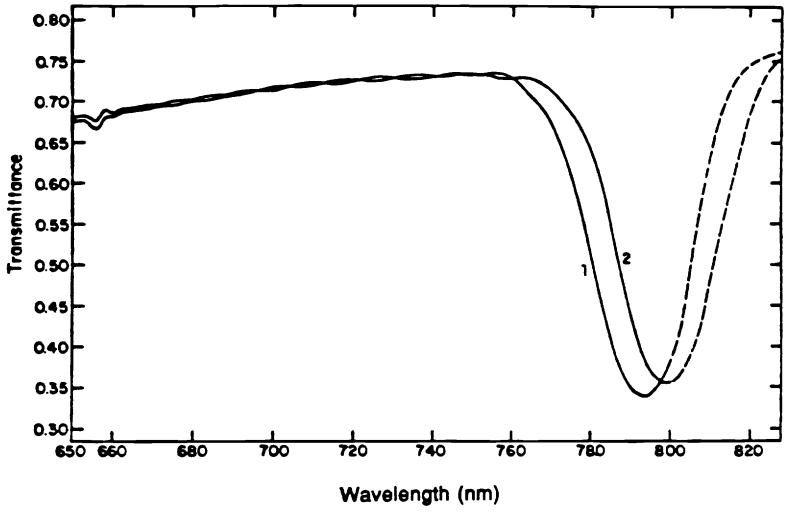

Figure 7. Transmittance as a function of Wavelength for a reflection filter at 0° incidence recorded at 130 mJ/cm^2.

3.1.5 *Holographic lenses in Dichromated Polyvinyl alcohol (DCPVA)*

(i). Development of DC-PVA for hologram recording

As mentioned earlier the maximum diffraction efficiency obtained for real-time recording with linear parallel polarized light at 488 nm is 18% (17). F.Ziping et.al.(18) developed the recorded holograms with a thermal treatment and obtained maximum

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diffraction efficiencies of ≈32%. We have tried a method of improving the final diffraction efficiency of the holograms (13). The development procedure is given elsewhere (Ref.24). The results of this study are illustrated in Fig.8. It was found that the maximum diffraction efficiency was 60% at a exposure level of 500 mJ/cm^2.

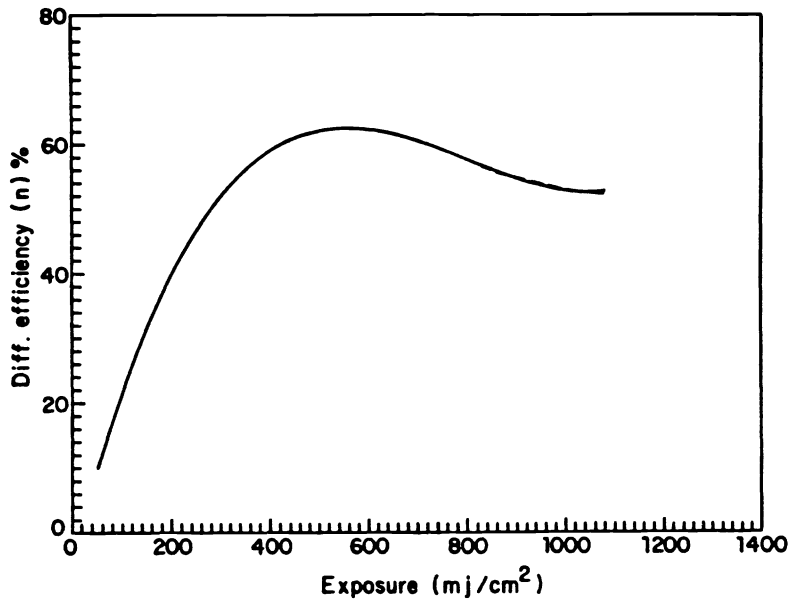

Figure 8. Diffraction as a function of exposure energy of a DCPVA transmission grating.

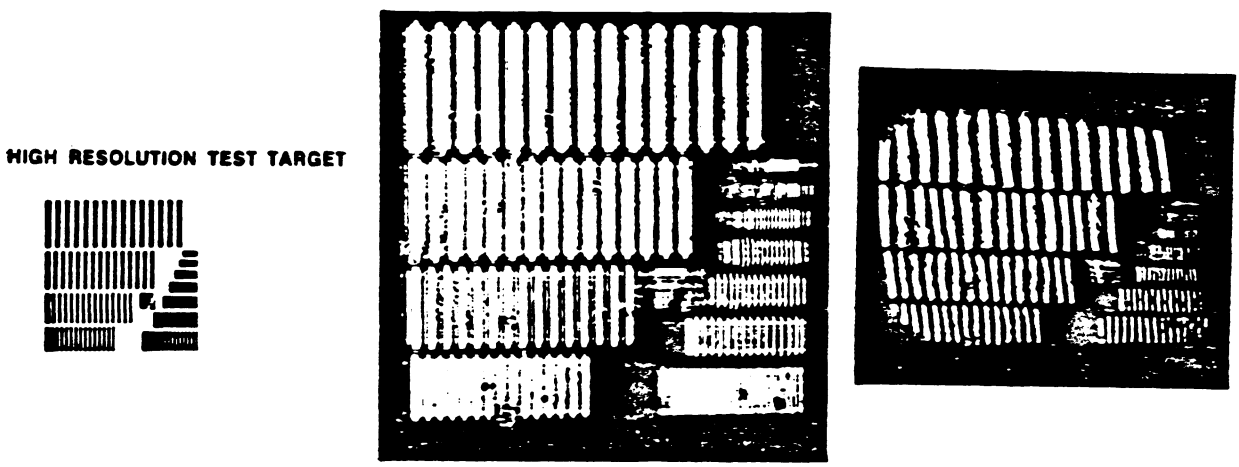

Figure 9. a) Ealing corporation high resolution chart, b) Tranmitted image , c) Reconstructed image using DCPVA holographic lens.

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(ii) *Holo-lenses in DCPVA*

Holographic lenses have been fabricated in DCPVA with focal length of 50 mm and aperture of 25 mm. To evaluate the performance of these lenses a Ealing corporation High resolution test target was used for imaging. The test chart, the transmitted image and the diffracted image from the Holo-lens are shown in Fig 10 a,b and c respectively. The distortion in the reconstructed image is mainly due to abberations introduced due to the shift in the wavelength of reconstruction.

CONCLUSIONS

Holographic filters and Holo-lenses were fabricated in Methylene blue sensitized Gelatin (MBG) and Dichromated Polyvinyl alcohol (DCPVA) and their characteristics studied. MBG filters can now be made with lower exposure energy (≈46 mJ/cm^2) and can be tuned to the required wavelength of reconstruction. High diffraction efficiency (≈80 %) has been acheived both in transmission and reflection holograms. DCPVA holograms exhibit efficiencies of 60% and have been used to fabricate holographic lenses.

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REFERENCES

1. T.A.Shankoff, "Phase holograms in dichromated gelatin", Appl.Opt. **7**, 2101 (1968).

2. B.J.Chang and C.D.Leonard, "Dichromated gelatin for the fabrication of Holographic optical elements":, Appl.Opt. **18**, 2407 (1979).

3. D.J.McCartney, D.B.Payne and S.S.Duncan, "Position-tunable holographic filters in Dichromated gelatin for use in single-mode fiber demultiplexers", Opt.Lett., **10**, 303 (1985).

4. T.Kubota, T.Ose, M.Sasaki, K.Honda, "Hologram formation with red light in methylene blue sensitized dichromated gelatin", Appl.Opt. **25**, 556 (1976).

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5. T.Kubota and T.Ose, "Lippmann color holograms recorded in Methylene blue sensitized dichromated gelatin", Opt. lett. **4**, 289 (1979).

6. A.Graube, "Dye sensitized dichromated gelatin for holographic optical element fabrication", Photo.Sci.Engg **22**, 37 (1978).

7. M.Akagi, "Spectral sensitization of Dichromated gelatin", Phot.Sci.Engg., **18**, 248 (1974).

8. R.Changkakoti and S.V.Pappu, "Towards optimum diffraction efficiency for methylene blue sensitized dichromated gelatin", Opt.Laser Tech. **21**, 259 (1989).

9. C.Solano, R.A.lessard and P.C.Roberge, "Methylene blue sensitized gelatin as a photosensitive medium for conventional and polarizing holography", Appl.Opt. **26**, 1989 (1987).

10. Nadia Capolla and Roger A.Lessard, "Processing of holograms recorded in methylene blue sensitized gelatin", Appl.Opt. **27**, 3008 (1988).

11. R.Changkakoti, S.S.C.Babu and S.V.Pappu, "Role of external electron donor in Methylene blue sensitized dichromated gelatin holograms: an experimental study", Appl.Opt. **27**, 324 (1988).

12. Jeff Blyth,"Methylene blue sensitized dichromated gelatin holograms: a new electron donor for their improved photsensitivity", Appl.Opt. **30**, 1598 (1991).

13. R.Changkakoti, Roger A.Lessard and J.J.A.Couture, "Dichromated PVA for the fabrication of holographic optical elements", OSA annual meeting, Technical Digest, 98, Nov (1990).

14. Roger. A.Lessard, Rupak Changkakoti and G.Manivannan, "Metal ion doped polymer system for real time holographic recording ", presented at the SPIE conference at San Diego, July 21-26 (1991).

15. G.Manivannan, Rupak Changkakoti and Roger A.Lessard, "Cr(IV) and Fe(III) doped polymer sustems as Real-time holographic recording materials", presented at the SPIE conference on Emerging Optoelectronic Technology at Bangalore,INDIA Dec. 18-20 (1991).

16. M.Sasaki, K.Honda and S.Kikuchi, "Studies on photosensitive dichromated materials", Report of the institute of industrial science , The University of Tokyo", **27**, Serial no 171, March 1978.

17. Sylviane Lelièvre and Jean J.A.Couture, "Dichromated polyvinyl alcohol films used as

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a novel polarization real time holographic recording material", Applied Optics **29**, 4384 (1990).

18. F.Ziping and Z.Jugin, "The study and application of dichromated-polyvinyl alcohol as real time holographic recording material", Guangxue Xuebao **4**, 1101 (1984).

19. Behzad Moslehi, Phil Harvey, Joel Ng, and Tomasz Jannson, "Fiber-optic wavelength-division multiplexing and demultiplexing using volume holographic gratings", Opt. letters **14**, 1088 (1989).

20. N.Capolla, R.A.Lessard, C.Carre and D.J.Lougnot, "Studies of the photoreduction of Methylene blue in gelatin solutions and films in view of improving the holographic recording process at 633 nm", Appl. Physics B **52**, 326 (1991).

21. N.Capolla and Roger A.Lessard,"Methylene blue sensitized gelatin used for the fabrication of holographic optical elements in the near-infrared", SPIE 1389, (1990).

22. N. Capolla and Roger A.Lessard, "Improving the recording speed of methylene blue sensitized gelatin by the addition of electron donors", submitted to applied optics.

23. H.Kogelnik, "Coupled wave theory for thick holograms", Bell Syst.Tech.Jl. **48**, 2909 (1969).

24. Roger A.Lessard, Rupak Changkakoti and G.Manivannan, "Recording materials based on vinyl polymers for Holography and Non-linear optics, presented at the SPIE conference on Emerging Optoelectronic Technology at Bangalore, India Dec 18-20 (1991).

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